6.9.4

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 10
1200 Sixth Avenue
Seattle, Washington 98101

IN REPLY
REFER TO: OEA-095 June 17, 1999

MEMORANDUM

SUBJECT: Bunker Hill, CLP Metals Analysis, Data Validation
Case: 26932
SDG: MJAH64

FROM: Laura Castrilli, Chemist
Quality Assurance and Data Unit, OEA

TO: Mary Kay Voytilla, Regional Project Manager
Office of Environmental Cleanup

CC: Bruce Woods, Region 10 CLP TPO
Jim Stefanoff, CH2M Hill

The following is a validation of ICP-AES and mercury analyses of five total and five dissolved water samples from the Bunker Hill project. The analyses were performed following the USEPA Contract Laboratory Program Statement of Work for Inorganics Analysis Multi-media, Multi-Concentration, ILM04.0. Analyses were conducted by Southwest Laboratories of Oklahoma, of Broken Arrow, Oklahoma. This validation was conducted for the following samples:

MJAH64	MJAH66	MJAH68	MJAH70	MJAH72
MJAH65	MJAH67	MJAH69	MJAH71	MJAH73

Data Qualifications

The following comments refer to the Southwest Laboratory's performance in meeting quality control specifications outlined in the *CLP Statement of Work (CLP-SOW) for Inorganic Analysis, rev. ILM04.0.* The comments presented herein are based on the information provided for the review.

1.0 Timeliness - Acceptable

The technical (40 CFR part 136) holding time from the date of collection for mercury in water is 28 days. The holding time for the remaining metals in water is 180 days. The samples were collected on 04/13/99. Mercury analyses were completed on 04/22/99. ICP-AES analyses were completed on 05/08/99.

2.0 Sample Preparation - Acceptable

The samples were prepared for mercury and ICP-AES analyses on 04/22/99.

3.0 Calibrations/Calibration Verifications - Acceptable

The samples were analyzed for mercury by CVAAS on 04/22/99. Initial calibration included one blank and five standards. The curve was linear with a correlation coefficient greater than 0.995.

The samples were analyzed by ICP-AES on 04/26/99 (main analyses), 05/07/99 (iron, manganese, and zinc dilutions), and 05/08/99 (zinc dilutions). The instrument was standardized according to the analytical method each day of analysis using one blank and a single calibration standard for each element.

All ICP-AES and CVAAS (mercury) calibrations were performed as required and met the acceptance criteria; therefore, no qualification was made on this basis.

Continuing calibration verifications (CCVs) are required before and after sample analysis and after every 10 samples during analysis. Mercury recoveries must be within 80-120%. Other metal recoveries must be within 90-110%. The frequency of analysis of CCVs was met. All ICP-AES and CVAAS (mercury) CCVs (initial and continuing) bracketing reported sample results met the recovery criteria; therefore, no qualification was made on this basis.

4.0 Laboratory Control Samples - Acceptable

Laboratory Control samples are digested and analyzed along with the samples to verify the efficiency of laboratory procedures. All recoveries associated with reported sample results met the acceptance criteria.

5.0 Blanks -

Procedural blanks were prepared with the samples to show potential contamination from the digestion or analytical procedure. If an analyte was found in the associated blank, the sample results were qualified if the analyte concentration was less than five times the analytical value in the blank.

Aluminum, calcium, chromium, iron, and zinc were detected in the preparation blank. Aluminum was detected in a couple ICP-AES continuing calibration blanks (CCBs). Magnesium had a negative value with an absolute value greater than the detection limit in one CCB. Based on blank contamination, chromium in samples MJAH65, MJAH66, MJAH67, MJAH70, MJAH72, and MJAH73 was qualified 'U', undetected.

All other sample results were greater than five times the associated

blank levels (or were already undetected) and were not qualified based on blank contamination.

6.0 ICP-AES Interference Check Sample -

The interference check sample (ICS) is analyzed by ICP-AES to verify interelement and background correction factors. Analysis is required at the beginning and end of each sample analysis run and recoveries must be between 80% and 120%. All ICS recoveries associated with reported sample results were within the recovery criterion.

The raw data for a number of samples had interfering levels of iron. Analytes for which iron is an interferent were qualified as follows:

- Selenium in samples MJAH65, MJAH67, MJAH70, and MJAH72 was qualified 'UJ', estimated detection limit (possible false negatives due to high iron) or 'J', estimated (possible low bias due to high iron). Selenium in two of the three ICS-A analyses bracketing these samples had negative results with absolute values greater than the detection limit.
- Vanadium in samples MJAH65, MJAH67, MJAH70, and MJAH72 was qualified 'UJ', estimated detection limit (possible false positives due to high iron). Vanadium in two of the three ICS-A analyses bracketing these samples had results greater than the detection limit.

Some of the samples required one or more dilution runs to report iron, manganese, and/or zinc results within the instrumental linear range. The raw data for all analytes were compared using the available dilutions to see if 1) cadmium, zinc, iron, and/or manganese levels in the undiluted samples were high enough that interelement corrections may not be sufficient for the analytes that were reported from the undiluted analyses or 2) a pattern of suppression or enhancement was evident. From this comparative study, no results were qualified due to suspected interference.

7.0 Duplicate Analysis - Acceptable

Duplicate analyses were done on sample MJAH64. Water duplicate results were within the ±20% Relative Percent Difference (RPD) or ±CRDL criteria for water results < 5 times the CRDL criteria.

8.0 Field Duplicate Analysis - Not Applicable

Field duplicate analysis for samples in this SDG was not indicated in the field collection documentation.

9.0 Matrix Spike Analysis -

Matrix spike sample analyses are done to provide information about the effect of the sample matrix on digestion and measurement methods. Matrix spike recovery must be within the limits of 75 - 125%.

Matrix spike analyses were done on sample MJAH64. All matrix spike recoveries were within the required QC limits, with the exception of selenium (51%). All selenium results were qualified 'J', estimated (possible low bias).

10.0 Graphite Furnace Atomic Absorption Spec (GFAAS) QC - Not Applicable -

GFAAS was not used for the analysis of these samples.

11.0 ICP-AES Serial Dilution - Acceptable

Sample MJAH64 was analyzed by ICP-AES serial dilution to check for potential interferences. All analytes which exceeded the minimum concentration criterion (50 times the IDL) agreed within the 10%D criteria.

12.0 Detection Limits - Acceptable

Sample results which fall below the instrument detection limit (IDL) are assigned the value of the instrument detection limit and the 'U' qualifier is attached. Contract Required Detection Limit (CRDL) standards are required to demonstrate a linear calibration curve near the CRDL. CRDL standards were run at the required frequency.

13.0 Overall Assessment of the Data

This validation of the data is based on the criteria outlined in the *National Functional Guidelines for Inorganic Data Review (02/94)*. Approximately 9% of the data was qualified based on blank contamination, interference, or matrix spike recovery. The data as qualified is acceptable for all purposes.

Below are the definitions for the National Functional Guidelines for Inorganic Data Review (02/94) qualifiers used when validating/qualifying data from Inorganic analysis.

DATA QUALIFIERS

U - The material was analyzed for, but was not detected above the level of the associated value. The associated value is either the sample quantitation limit or the sample detection limit.

J - The associated value is an estimated quantity.

R - The data are unusable. (Note: Analyte may or may not be present.)

UJ - The material was analyzed for, but was not detected. The associated value is an estimate and may be inaccurate or imprecise.

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INORGANIC ANALYSES DATA SHEET

EPA SAMPLE NO.

MJAH64 ___

Lab Name: SOUTHWEST_LAB_OF_OKLAHOMA Contract: 68-D5-0136

Lab Code: SWOK__ Case No.: 26932_ SAS No.: _____ SDG No.: MJAH64

Matrix (soil/water): WATER Lab Sample ID: 38058.01

Level (low/med): LOW__ Date Received: 04/14/99

% Solids: __0.0

Concentration Units (ug/L or mg/kg dry weight): UG/L_

CAS No.	Analyte	Concentration	C	Q	M
7429-90-5	Aluminum	291			P
7440-36-0	Antimony	3.0	U		P
7440-38-2	Arsenic	37.7			P
7440-39-3	Barium	17.2	B		P
7440-41-7	Beryllium	1.0	U		P
7440-43-9	Cadmium	40.9			P
7440-70-2	Calcium	8620			P
7440-47-3	Chromium	1.0	U		P
7440-48-4	Cobalt	18.3	B		P
7440-50-8	Copper	25.5			P
7439-89-6	Iron	33200			P
7439-92-1	Lead	1310			P
7439-95-4	Magnesium	5930			P
7439-96-5	Manganese	7570			P
7439-97-6	Mercury	0.10	U		CV
7440-02-0	Nickel	19.0	B		P
7440-09-7	Potassium	1290	B		P
7782-49-2	Selenium	3.0	U	~~N~~ J	P
7440-22-4	Silver	1.0	U		P
7440-23-5	Sodium	1020	B		P
7440-28-0	Thallium	5.0	U		P
7440-62-2	Vanadium	1.0	U		P
7440-66-6	Zinc	16200			P

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INORGANIC ANALYSES DATA SHEET

EPA SAMPLE NO.

MJAH65

Lab Name: SOUTHWEST_LAB_OF_OKLAHOMA Contract: 68-D5-0136

Lab Code: SWOK Case No.: 26932 SAS No.: SDG No.: MJAH64

Matrix (soil/water): WATER Lab Sample ID: 38058.02

Level (low/med): LOW Date Received: 04/14/99

% Solids: 0.0

Concentration Units (ug/L or mg/kg dry weight): UG/L

CAS No.	Analyte	Concentration	C	Q	M
7429-90-5	Aluminum	35100			P
7440-36-0	Antimony	3.0	U		P
7440-38-2	Arsenic	1380			P
7440-39-3	Barium	12.2	B		P
7440-41-7	Beryllium	9.6			P
7440-43-9	Cadmium	2420			P
7440-70-2	Calcium	45000			P
7440-47-3	Chromium	6.6	~~B~~	U	P
7440-48-4	Cobalt	244			P
7440-50-8	Copper	853			P
7439-89-6	Iron	1230000			P
7439-92-1	Lead	1310			P
7439-95-4	Magnesium	96700			P
7439-96-5	Manganese	116000			P
7439-97-6	Mercury	0.11	B		CV
7440-02-0	Nickel	182			P
7440-09-7	Potassium	671	B		P
7782-49-2	Selenium	3.0	U	~~NJ~~	P
7440-22-4	Silver	3.0	B		P
7440-23-5	Sodium	947	B		P
7440-28-0	Thallium	49.4			P
7440-62-2	Vanadium	5.0	~~B~~	UJ	P
7440-66-6	Zinc	860000			P

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INORGANIC ANALYSES DATA SHEET

EPA SAMPLE NO.
MJAH66

Lab Name: SOUTHWEST_LAB_OF_OKLAHOMA Contract: 68-D5-0136

Lab Code: SWOK Case No.: 26932 SAS No.: _____ SDG No.: MJAH64

Matrix (soil/water): WATER Lab Sample ID: 38058.03

Level (low/med): LOW Date Received: 04/14/99

% Solids: 0.0

Concentration Units (ug/L or mg/kg dry weight): UG/L

CAS No.	Analyte	Concentration	C	Q	M
7429-90-5	Aluminum	1780			P
7440-36-0	Antimony	3.0	U		P
7440-38-2	Arsenic	26.7			P
7440-39-3	Barium	20.8	B		P
7440-41-7	Beryllium	1.0	U		P
7440-43-9	Cadmium	142			P
7440-70-2	Calcium	11400			P
7440-47-3	Chromium	1.0	~~B~~ U		P
7440-48-4	Cobalt	10.4	B		P
7440-50-8	Copper	47.1			P
7439-89-6	Iron	23600			P
7439-92-1	Lead	266			P
7439-95-4	Magnesium	20400			P
7439-96-5	Manganese	11800			P
7439-97-6	Mercury	0.10	U		CV
7440-02-0	Nickel	11.0	B		P
7440-09-7	Potassium	630	B		P
7782-49-2	Selenium	3.0	U	~~N~~ J	P
7440-22-4	Silver	1.0	U		P
7440-23-5	Sodium	980	B		P
7440-28-0	Thallium	5.0	U		P
7440-62-2	Vanadium	1.0	U		P
7440-66-6	Zinc	36600			P

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INORGANIC ANALYSES DATA SHEET

EPA SAMPLE NO.

MJAH67 ___

Lab Name: SOUTHWEST_LAB_OF_OKLAHOMA Contract: 68-D5-0136

Lab Code: SWOK__ Case No.: 26932_ SAS No.: _____ SDG No.: MJAH64

Matrix (soil/water): WATER Lab Sample ID: 38058.04

Level (low/med): LOW__ Date Received: 04/14/99

% Solids: __0.0

Concentration Units (ug/L or mg/kg dry weight): UG/L_

CAS No.	Analyte	Concentration	C	Q	M
7429-90-5	Aluminum	65700			P
7440-36-0	Antimony	3.0	U		P
7440-38-2	Arsenic	2620			P
7440-39-3	Barium	14.7	B		P
7440-41-7	Beryllium	17.1			P
7440-43-9	Cadmium	4400			P
7440-70-2	Calcium	69700			P
7440-47-3	Chromium	10.4		U	P
7440-48-4	Cobalt	442			P
7440-50-8	Copper	1650			P
7439-89-6	Iron	2500000			P
7439-92-1	Lead	1370			P
7439-95-4	Magnesium	132000			P
7439-96-5	Manganese	225000			P
7439-97-6	Mercury	0.23			CV
7440-02-0	Nickel	327			P
7440-09-7	Potassium	618	B		P
7782-49-2	Selenium	18.4		~~N~~ J	P
7440-22-4	Silver	1.2	B		P
7440-23-5	Sodium	911	B		P
7440-28-0	Thallium	136			P
7440-62-2	Vanadium	11.5	~~B~~	UJ	P
7440-66-6	Zinc	1590000			P

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EPA SAMPLE NO.
MJAH68 ___

Lab Name: SOUTHWEST_LAB_OF_OKLAHOMA Contract: 68-D5-0136

Lab Code: SWOK__ Case No.: 26932_ SAS No.: _____ SDG No.: MJAH64

Matrix (soil/water): WATER Lab Sample ID: 38058.05

Level (low/med): LOW__ Date Received: 04/14/99

% Solids: __0.0

Concentration Units (ug/L or mg/kg dry weight): UG/L_

CAS No.	Analyte	Concentration	C	Q	M
7429-90-5	Aluminum	1780			P
7440-36-0	Antimony	3.0	U		P
7440-38-2	Arsenic	28.3			P
7440-39-3	Barium	20.8	B		P
7440-41-7	Beryllium	1.0	U		P
7440-43-9	Cadmium	141			P
7440-70-2	Calcium	11300			P
7440-47-3	Chromium	1.0	U		P
7440-48-4	Cobalt	10.5	B		P
7440-50-8	Copper	44.9			P
7439-89-6	Iron	22900			P
7439-92-1	Lead	276			P
7439-95-4	Magnesium	20100			P
7439-96-5	Manganese	11600			P
7439-97-6	Mercury	0.10	U		CV
7440-02-0	Nickel	10.8	B		P
7440-09-7	Potassium	648	B		P
7782-49-2	Selenium	3.0	U	~~N~~ J	P
7440-22-4	Silver	1.0	U		P
7440-23-5	Sodium	905	B		P
7440-28-0	Thallium	5.0	U		P
7440-62-2	Vanadium	1.0	U		P
7440-66-6	Zinc	36600			P

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INORGANIC ANALYSES DATA SHEET

EPA SAMPLE NO.
MJAH69 ___

Lab Name: SOUTHWEST_LAB_OF_OKLAHOMA Contract: 68-D5-0136

Lab Code: SWOK__ Case No.: 26932_ SAS No.: _____ SDG No.: MJAH64

Matrix (soil/water): WATER Lab Sample ID: 38058.06

Level (low/med): LOW__ Date Received: 04/14/99

% Solids: __0.0

Concentration Units (ug/L or mg/kg dry weight): UG/L_

CAS No.	Analyte	Concentration	C	Q	M
7429-90-5	Aluminum	272			P
7440-36-0	Antimony	3.0	U		P
7440-38-2	Arsenic	20.5			P
7440-39-3	Barium	18.2	B		P
7440-41-7	Beryllium	1.0	U		P
7440-43-9	Cadmium	40.7			P
7440-70-2	Calcium	8330			P
7440-47-3	Chromium	1.0	U		P
7440-48-4	Cobalt	18.0	B		P
7440-50-8	Copper	27.0			P
7439-89-6	Iron	27200			P
7439-92-1	Lead	1260			P
7439-95-4	Magnesium	5760			P
7439-96-5	Manganese	7460			P
7439-97-6	Mercury	0.10	U		CV
7440-02-0	Nickel	18.8	B		P
7440-09-7	Potassium	1340	B		P
7782-49-2	Selenium	3.0	U	~~N~~ J	P
7440-22-4	Silver	1.0	U		P
7440-23-5	Sodium	1100	B		P
7440-28-0	Thallium	5.0	U		P
7440-62-2	Vanadium	1.0	U		P
7440-66-6	Zinc	15300			P

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INORGANIC ANALYSES DATA SHEET

EPA SAMPLE NO.

MJAH70 ___

Lab Name: SOUTHWEST_LAB_OF_OKLAHOMA Contract: 68-D5-0136

Lab Code: SWOK__ Case No.: 26932_ SAS No.: _____ SDG No.: MJAH64

Matrix (soil/water): WATER Lab Sample ID: 38058.07

Level (low/med): LOW__ Date Received: 04/14/99

% Solids: __0.0

Concentration Units (ug/L or mg/kg dry weight): UG/L_

CAS No.	Analyte	Concentration	C	Q	M
7429-90-5	Aluminum	35800			P
7440-36-0	Antimony	3.0	U		P
7440-38-2	Arsenic	1390			P
7440-39-3	Barium	13.1	B		P
7440-41-7	Beryllium	9.7			P
7440-43-9	Cadmium	2470			P
7440-70-2	Calcium	46200			P
7440-47-3	Chromium	3.1	~~B~~	U	P
7440-48-4	Cobalt	248			P
7440-50-8	Copper	875			P
7439-89-6	Iron	1220000			P
7439-92-1	Lead	1390			P
7439-95-4	Magnesium	98700			P
7439-96-5	Manganese	116000			P
7439-97-6	Mercury	0.10	U		CV
7440-02-0	Nickel	188			P
7440-09-7	Potassium	637	B		P
7782-49-2	Selenium	3.0	U	~~N~~ J	P
7440-22-4	Silver	1.1	B		P
7440-23-5	Sodium	1060	B		P
7440-28-0	Thallium	48.2			P
7440-62-2	Vanadium	6.3	~~B~~	UJ	P
7440-66-6	Zinc	870000			P

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INORGANIC ANALYSES DATA SHEET

EPA SAMPLE NO.
MJAH71 ___

Lab Name: SOUTHWEST_LAB_OF_OKLAHOMA Contract: 68-D5-0136

Lab Code: SWOK__ Case No.: 26932_ SAS No.: _____ SDG No.: MJAH64

Matrix (soil/water): WATER Lab Sample ID: 38058.08

Level (low/med): LOW__ Date Received: 04/14/99

% Solids: __0.0

Concentration Units (ug/L or mg/kg dry weight): UG/L_

CAS No.	Analyte	Concentration	C	Q	M
7429-90-5	Aluminum	1800			P
7440-36-0	Antimony	3.0	U		P
7440-38-2	Arsenic	11.9			P
7440-39-3	Barium	27.8	B		P
7440-41-7	Beryllium	1.0	U		P
7440-43-9	Cadmium	145			P
7440-70-2	Calcium	11700			P
7440-47-3	Chromium	1.0	U		P
7440-48-4	Cobalt	10.7	B		P
7440-50-8	Copper	45.4			P
7439-89-6	Iron	17600			P
7439-92-1	Lead	263			P
7439-95-4	Magnesium	20600			P
7439-96-5	Manganese	11900			P
7439-97-6	Mercury	0.10	U		CV
7440-02-0	Nickel	11.4	B		P
7440-09-7	Potassium	611	B		P
7782-49-2	Selenium	3.0	U	~~N~~ J	P
7440-22-4	Silver	1.0	U		P
7440-23-5	Sodium	1030	B		P
7440-28-0	Thallium	5.0	U		P
7440-62-2	Vanadium	1.0	U		P
7440-66-6	Zinc	36600			P

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INORGANIC ANALYSES DATA SHEET

EPA SAMPLE NO.
MJAH72 ___

Lab Name: SOUTHWEST_LAB_OF_OKLAHOMA Contract: 68-D5-0136

Lab Code: SWOK__ Case No.: 26932_ SAS No.: _____ SDG No.: MJAH64

Matrix (soil/water): WATER Lab Sample ID: 38058.09

Level (low/med): LOW__ Date Received: 04/14/99

% Solids: __0.0

Concentration Units (ug/L or mg/kg dry weight): UG/L_

CAS No.	Analyte	Concentration	C	Q	M
7429-90-5	Aluminum	66000			P
7440-36-0	Antimony	3.0	U		P
7440-38-2	Arsenic	2610			P
7440-39-3	Barium	16.4	B		P
7440-41-7	Beryllium	17.2			P
7440-43-9	Cadmium	4390			P
7440-70-2	Calcium	69700			P
7440-47-3	Chromium	11.8		U	P
7440-48-4	Cobalt	444			P
7440-50-8	Copper	1650			P
7439-89-6	Iron	2460000			P
7439-92-1	Lead	1390			P
7439-95-4	Magnesium	131000			P
7439-96-5	Manganese	222000			P
7439-97-6	Mercury	0.10	U		CV
7440-02-0	Nickel	327			P
7440-09-7	Potassium	617	B		P
7782-49-2	Selenium	24.3		~~N~~ J	P
7440-22-4	Silver	1.8	B		P
7440-23-5	Sodium	1140	B		P
7440-28-0	Thallium	132			P
7440-62-2	Vanadium	10.6	~~B~~	UJ	P
7440-66-6	Zinc	1600000			P

4/26/17/99

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INORGANIC ANALYSES DATA SHEET

EPA SAMPLE NO.
MJAH73 ___

Lab Name: SOUTHWEST_LAB_OF_OKLAHOMA Contract: 68-D5-0136

Lab Code: SWOK__ Case No.: 26932_ SAS No.: _____ SDG No.: MJAH64

Matrix (soil/water): WATER Lab Sample ID: 38058.10

Level (low/med): LOW__ Date Received: 04/14/99

% Solids: __0.0

Concentration Units (ug/L or mg/kg dry weight): UG/L_

CAS No.	Analyte	Concentration	C	Q	M
7429-90-5	Aluminum	1820			P
7440-36-0	Antimony	3.0	U		P
7440-38-2	Arsenic	6.4	B		P
7440-39-3	Barium	27.8	B		P
7440-41-7	Beryllium	1.0	U		P
7440-43-9	Cadmium	147			P
7440-70-2	Calcium	11800			P
7440-47-3	Chromium	1.0	B		P
7440-48-4	Cobalt	11.1	B		P
7440-50-8	Copper	46.6			P
7439-89-6	Iron	16000			P
7439-92-1	Lead	267			P
7439-95-4	Magnesium	21000			P
7439-96-5	Manganese	12100			P
7439-97-6	Mercury	0.10	U		CV
7440-02-0	Nickel	11.6	B		P
7440-09-7	Potassium	680	B		P
7782-49-2	Selenium	3.0	U	~~NJ~~	P
7440-22-4	Silver	1.0	U		P
7440-23-5	Sodium	1070	B		P
7440-28-0	Thallium	5.0	U		P
7440-62-2	Vanadium	1.0	U		P
7440-66-6	Zinc	36200			P

06/17/99

Color Before: COLORLESS Clarity Before: CLEAR_ Texture: _____

Color After: COLORLESS Clarity After: CLEAR_ Artifacts: _____

Comments:

FORM I - IN

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